In-situ BCDI of polar nanoregions in barium titanate nanocrystals during phase transition (MA-4704)

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In a 4-day experiment at ID01, we performed BCDI measurements of Barium Titanate (BTO) nanocrystals as a function of temperature. We bonded the crystals onto silicon substrates using the TEOS procedure. This worked well: even with the full coherent EBS beam of ID01, we found the position and angles of all crystals examined to be unaffected by radiation. These were installed in a furnace on the beamline which was able to stabilise the temperature within +/-50mK.

Long macro scans were performed to keep the selected crystals centered in the beam while heating them from 100^{o}C to 160^{o}C and back to examine the effects of the tetragonal to cubic phase transition, which occurs at 120^{o}C in the bulk. We measured Bragg peaks on the {110} powder ring, but some of these are 110 and others are 101, the two being split in the low temperature tetragonal phase. Four separate crystals were tracked on consecutive nights and all four gave good looking 3D coherent diffraction patterns. One of them showed three subgrains in the integrated rocking curve, which collapsed to a single peak at 140^{o}C suggesting that the phase transition was there. Upon cooling, the 3-peak structure reappeared at 120^{o}C indicating hysteresis. The elevated transition temperature is generally expected for nanoparticles because of strain and large surface area. Hysteresis effects are common at first order phase transitions and may indicate slow transition kinetics. Time dependence was not checked systematically, but at both 140^{o}C and 145^{o}C, three successive scans were made, which show progressive loss of the 3-peak structure, as seen in the figure.

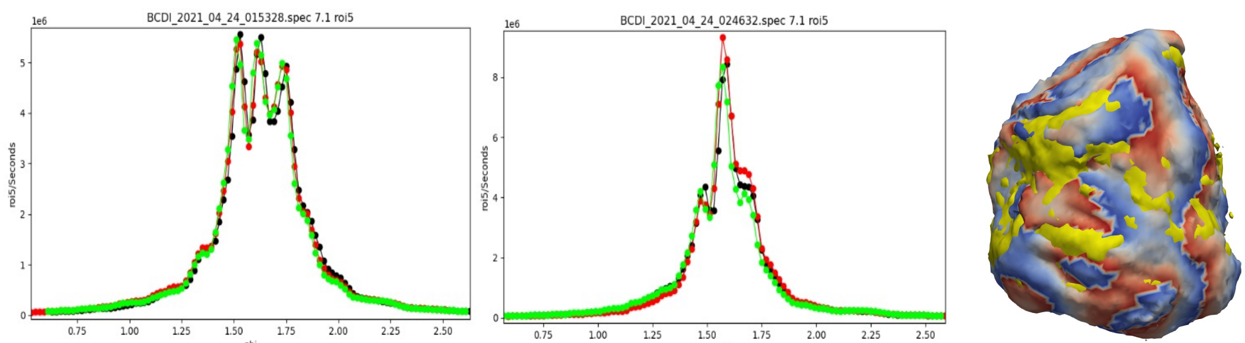

Figure. Integrated rocking curves of a single 300nm BTO nanocrystal at 130^{o}C and 140^{o}C (left and centre). The three traces are sequential in time. The BCDI coherent diffraction patterns 115^{o}C and 150^{o}C have been inverted to images which are overlaid in the right panel.

Reconstructions of the data have begun. The BTO diffraction patterns seem to reconstruct reliably and give reproducible crystal shapes, sometimes facetted. The next step will be to look at the internal phase structure in the images which will provide evidence of tetragonal domain formation. Previous results have shown the appearance of domain walls along {110} directions. Facetted crystals are better for this exercise because they can be indexed to give the full 3D crystal orientation, even though only one Bragg peak is measured.

During the daytime, we took off the BTO sample to characterise some other samples, which were part of another accepted proposal, delayed to later in summer 2021 because of COVID and the need to do some Laue indexing before measurement. These were Pd nanocrystals, dewetted from a thin film then implanted with 1 MeV Kr ions at temperature of 500^{o}C at fluence (dose) of 3.0×10^{16} and 4.5×10^{16} Kr ions/cm^2 respectively. We got 3-6 crystals measured from each of the two different doses along with baseline pristine (unimplanted) samples. The diffraction patterns showed correct fringes and flares and the reconstructions looked like they converged well. Further strain analysis will help us determine and understand the Kr ion irradiation induced damage as function of dose in Pd nanocrystals. Pd nanocrystals serve as model materials for studying effect of ion irradiation in metallic systems as they are single component materials with well-defined crystalline peaks suitable for elucidating strain.